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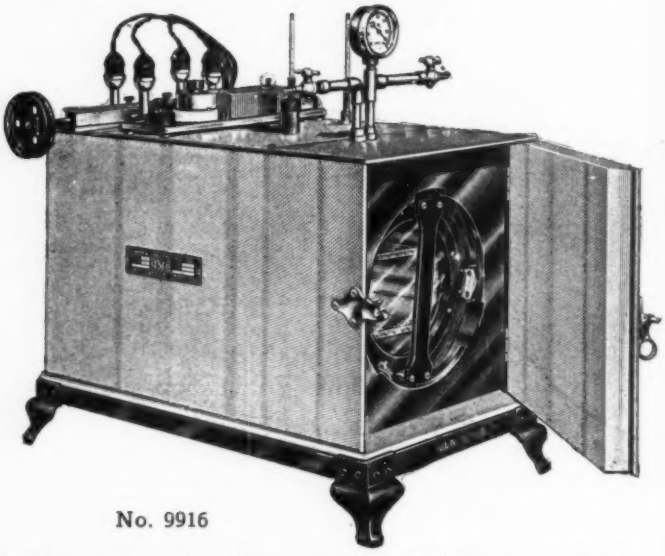

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VOL. III OCTOBER, 1926 No. 10

EDITORIAL

THE COTTON CROP AND THE OIL MILLS

With the statistician reporting the largest cotton crop on record in this country, one wonders what has become of the Boll Weevil which only a few short years ago led pessimists to believe that cotton would soon be exterminated and cotton oil mills and refineries reduced to piles of useless machinery.

Whether the bumper crop is due to excess planting to overcome the ravages of the weevil, or whether it results from unusually favorable weather conditions digging off of the weevils, the fact remains that the oil mills of the country can look ahead to unprecedented supplies of seed and reasonable prices which will enable them to operate at a good profit.

The reported good condition of the seed means good crude with small refining losses and high quality oils.

The large spread in price between hog lard and the splendid vegetable shortenings now on the market argues well for the future prospects of the refineries. Cheap seed should mean cheap cake and meal, and cotton seed meal being the cheapest source of protein, large quantities of meal should find its way to the cattle feeders.

The good quality of this year's seed should greatly help the increased use of meal. The oil mills have it in their hands to put the meal business on a better basis than in the past. Perhaps for local use the mixing of large quantities of hull with the meal may be good practice, if the price is made accordingly; but for distant shipments, it is suicide. The cattle feeders of the north buy cottonseed meal for protein. They do not want to pay freight on hulls. They have plenty of roughage without importing any.

This year's good seed crop offers splendid opportunities to work out efficient methods for grading seed and paying for what it is worth rather than fictitious prices forced by short sighted competition. In trading in oil and factory control of refineries the time is ripe to apply exact methods of chemical analysis in place of the crude refining tests used in the past. These served their purpose in the early days when there was little exact knowledge; but in this era, industry is ruled by exact science.

COMMERCIAL SOLVENTS FOR SOLVENT EXTRACTION

By LOUIS C. WHITON

Activity in connection with the solvent extraction of oil seeds has been particularly marked, of late, in the United States. This method has been widely and successfully adopted in Europe for the last ten or twenty years but comparatively little is known in the United States in connection with the practical application of different solvents. The use of the proper solvent is essential for the successful operation of such a plant, and in the past 25 years solvent extraction plants for oil seeds in the United States have frequently been unsuccessful, either because of the inability at the time they were constructed to obtain a proper solvent or because of ignorance as to which to use. This condition no longer obtains and a variety of useful solvents may without difficulty be procured in the United States, upon the open market.

This paper will discuss the physical constants of the most important of the solvents and their usefulness in commercial application as judged from experience in solvent extractor plants in the United States and abroad.

Requisites of a Solvent

The first requisite of a solvent for commercial extraction of oil seeds is evidently that it dissolve the oil. Certain products described below are excellent for this purpose but have the additional tendency to dissolve color from the seed, which is an undesirable feature.

The second condition is that the solvent be easily vaporized and homogeneous in composition, so that with facility, it can be removed completely by steaming from the oil and the extracted residue.

A third requisite is that the entire solvent distill at a temperature below 212°, in spite of the fact that a higher boiling solvent will vaporize at a lower temperature in the presence of water vapor. "Heavy Ends" are extremely difficult to remove from the residual meal, and the attempt to use solvents possessing them, in the past, has given the impression to those not familiar with modern extractor practice that the oil and meal cannot be entirely deodorized from the solvent. The hundreds of plants in operation primarily in Europe have demonstrated conclusively that this is easily accomplished in a correctly designed system and provided a solvent with the proper boiling range is used.

The fourth requisite is that there be no "light ends." These products are difficult to condense, will eventually be lost and, in the case of inflammable solvents, increase the fire risk. In addition, they may be injurious to the health of the operators as uncondensed vapor in the atmosphere of the plant, although in a properly operated solvent extraction there should be no odor whatever of solvent.

The fifth consideration is the relative fire hazard. The explosion risk is negligible, since in reality there are very rarely explosions within an extractor, since the exact quantity of air and solvent vapor must be encountered. Such explosions as have occurred have generally been steam explosions, caused by too great pressure of steam within a still or extractor. This can evidently be obviated by proper design.

Description of Various Solvents

Solvents may be classed in one of three categories:

Inflammable Solvents:	Petroleum Ether
	Gasoline (motor fuel)
	Special cut of gasoline
	Ether
	Benzol
Semi-Inflammable Solvents:	Ethylene Dichloride
	Trichlorethylene 75%—Benzol 25% Mixture
	Carbon tetrachloride 30%—Gasoline cut 70% Mixture
Non-Inflammable Solvents:	Trichlorethylene
	Carbon tetrachloride

Petroleum Ether: For use in commercial extraction plants, the product which is known in the laboratory as petroleum ether is not interesting except in special cases. Its boiling point is so low that it is difficult to condense and therefore the loss is unduly high. In view of its high cost, this removes it from practical consideration for the extraction of oil seeds.

Gasoline: Gasoline such as is used for motor fuel is not suitable for the extraction of oil bearing seeds without further refinement. The term "gasoline" is used somewhat loosely for any cut from low boiling petroleum products. The terms naphtha and benzine are also frequently employed but are merely trade names for slightly different types of gasoline.

The unsuitability of motor fuel gasoline for the extraction of edible oil will be appreciated by an examination of the average analysis of 1,049 samples collected by the Texas Company, and given below.

Per cent by Vol.	Av. 1049 samples	Govt. Specifications	Sample A Motor fuel	Sample B special	Sample C special
Initial B. Pt	114° F	141	88	107	80
20%	208	221	187	...	..
50%	277	284	260	...	..
90%	378	392	355	...	..
End Point	431	437	396	328	212

There has also been included three samples of gasoline products obtainable upon the open market, and the U. S. Government specification for a motor fuel.

It will be seen that the "heavy ends" of gasoline in extracted oil or meal would require intensive steaming which is not only wasteful of steam but is the principal cause of an extracted meal or oil possessing a solvent odor. It may be considered axiomatic therefore that for practical solvent extraction plant operation, no solvent should be used if any portion of it boils over 212°F. It might also be added that it is not advantageous to have any product boiling below 125° F.

Special Cut of Gasoline: On the European market there is easily obtainable a special cut of gasoline made from the American product by the refineries in Europe with the following specifications:

Boiling Point	122°F
2%	140°
97%	194°
End Point	200°

This product is the ideal solvent from the standpoint of the quality of oil produced. It has been produced in the United States at individual extraction plants from good quality gasoline similar to Sample A listed above. Although the boiling points indicated for the sample would not appear to make it possible to obtain half of this gasoline in the form of a rectified solvent similar to the special cut mentioned above, it nevertheless is a fact in practise, as the writer has observed from his own experience. It must be remembered that the boiling points shown above were determined in the laboratory in a simple distillation apparatus without a rectifying column, and are therefore empiric.

It might be of interest at this point to briefly describe a simple and inexpensive type of rectifying apparatus which produced this product.* A kettle still equipped with steam coils and direct steam was surmounted by a 8" pipe 6' long which was filled with small glass rings, thus resembling a Hempel column. These glass rings were made from glass tubing cut to short lengths and may be obtained commercially at a cost of approximately $25.00 per cu. ft. A dephlegmator was attached to the rectifying column and an excellent clear-cut product produced which was even better in quality, in the opinion of the writer, than could have been obtained with a bell and collar type of distillation column occupying the same space or costing the same to install.

Ether: Ether is not a solvent suitable for the extraction of oil

* Chemical and Metallurgical Engineering, January 3, 1923.

seeds because of its high cost and the difficulty experienced in condensing it without undue loss.

Benzol C_6H_6:

Boiling Point	176.0 °F
Freezing Point	42 °F
Specific Gravity at 68°F	0.878
Latent Heat	92.9 Cal.
Boiling Pt. Benzol—water mixture	155 °F

This product is one of the easiest to obtain and most satisfactory solvent for many substances. Its disadvantage lies principally in its tendency, in common with the other aromatic series of solvents, to dissolve some color from certain seeds, and thus produce a darker oil than that obtainable with the special gasoline cut mentioned above.

The price is approximately the same as gasoline and its distillation range should not be more than 4°F, one of the points being its boiling point of 176°F. It is therefore an extremely pure compound which is easy to distill and condense. It is consequently extremely easy to eliminate all traces of this solvent from the oil and meal. It should be thiophene free.[1]

It is possible that a satisfactory and very cheap solvent can be derived from the "Light Oil" manufactured at the benzol plants of coke-oven and gas works. The cut known as "raw benzol" boils between 140°F. and 230°F. It is only during the last four hours of a twenty hour distillation in the raw benzol still that any products are produced boiling over 195°F. Therefore it would be possible to cut the distillation at this point and to throw all of the products above 195°F. into the raw toluol fraction. This would produce a solvent with the proper boiling point, but the degree of necessary refining would have to be determined from experience of extracting with the above product. The writer does not know of instances where this product has been employed for extraction, but offers it as a suggestion as a possible low cost solvent.

Semi-Inflammable Solvents

Ethylene Dichloride[2] $CH_2Cl\text{-}CH_2Cl$.

Flash point (open cup)	60°F
Fire point	90°F
Specific Gravity at 68°F	1.2569
Boiling Pt.	182°F
Freezing Pt.	—32°F
Specific Heat	0.3054 at 86°F
Heat of vaporisation	157.5 BTU per lb. at 32°F

This compound has been variously known as "Dutch Liquid"

[1] To test for this product qualitatively, place a few crystals of isatin in a shallow porcelain dish previously rinsed with sulphuric acid. Add 0.5 cc. sulphuric acid and 5 cc. benzol. Cover with watch glass and allow to stand 30 minutes. If blue rings occur around the isatin, thiophene is present.—Lunge-Kohler, **Steinkohlenteer & Ammoniak, Vol. 1.**

[2] The information contained herein is based upon that furnished through the courtesy of the Carbide & Carbon Chemical Co., and that contained in the article on ethylene dichloride in Chemical & Metallurgical Engineering, Vol. 25, No. 22, Nov. 30, 1921.

"Elaychloride" "Ethylene Chloride" or by the more exact term "1, 2-dichlorethylene."

This is intermediate between inflammable products such as have been described above and the non-inflammable or fire extinguisher products such as trichlorethylene and carbon tetrachloride over which it is claimed it has the advantage of hydrolysing to a less extent. Its inflammability is low since it requires a concentration in air of 6.2 per cent before the lower explosive limit is reached, whereas the lower explosive limit of ether in air is 1.71 per cent, benzol 1.41 per cent and an average gasoline 1.5 per cent. Looked at from one point of view, it may therefore be considered as having one-quarter the tendency to explode as these inflammable products. However it should be noted that ethylene dichloride air mixture cannot be exploded by a spark but requires an open flame at least 1 or 2 inches in length to start the explosion which is a feeble one. The product possesses a characteristic odor similar to chloroform and trichlorethylene and it is quite certain that its presence in air would be detected by the sense of smell long before its concentration approaches the 6 per cent mark. Its inflammability is not great, since, although it will burn, a draft of air created by the heat is sufficient to extinguish the flame. Due to the fact that it is heavier than water, it may be extinguished by water in contradistinction to benzol or gasoline.

With a solvent such as ethylene dichloride, the writer does not believe that there is any real fire or explosion hazard in connection with any recognized widely-adopted extraction system. However it would appear as an important step on the part of the manufacturers of this product for them to settle the matter definitely from the insurance underwriter's standpoint, since the principal advantage of an expensive solvent such as this (costing $0.10 per pound, or $1.07 per gallon) is the lowest insurance rate that should be obtainable. Also the authority should be obtained to utilize such a product within the limits of large cities.

Trichlorethylene 75 Per Cent—Benzol 25 Per Cent Mixture: This mixture is a semi-inflammable and similar in this respect to ethylene-dichloride. Its main advantage lies in the fact that the cost of benzol is approximately one-quarter that of trichlorethylene and therefore the cost of the mixture is less than the pure trichlorethylene. The writer does not know of any extraction plants using this mixture.

Carbon-tetrachloride 30 Per Cent—Special Gasoline Cut 70 Per Cent Mixture: This product is similar to the mixture mentioned above. The tendency for the carbon tetrachloride to hydrolyze precluded it from use with steel or iron apparatus. Margosches and Braunlich[1] and Merz[2]

[1] Margosches. Der Tetrachlorkohlenstoff, Stuttgart, 1905.
[2] Fritsch, Fabrication et Raffinage des Huiles Vegetales, 1922.
[3] Much of the technical data on this substance has been supplied through the courtesy of Roessler and Hasslacher Co.

maintain that such a mixture is impossible to use commercially because of its tendency to fractionally separate during distillation.

Non-Inflammable Solvents

Trichlorethylene[3] $CHCl:CCl_2$

Boiling Point	190.4°F
Freezing Point	94°F
Vapor tension at 77°F	56 mm
Latent Heat (Cal.)	56.6 Cal.
Specific Heat at 64.4°F	0.233
Specific Gravity at 77°F	1.460

This product may be classed as absolutely non-inflammable and may be used as a fire-extinguisher liquid. It is used to a considerable extent for the extraction of various materials including oil-seeds. Because of its gravity the current of solvent should, if possible, pass upward through the mass of seed since the solution of oil and solvent has a slightly lower specific gravity than the pure solvent and therefore has a tendency to rise to the top.

It formerly was manufactured abroad only and the duty of 35 per cent brought its cost to over $0.11 per pound. It is now being produced in the United States, and is sold at a price (at the present writing) between 8½c and 9c per pound. This makes its cost per gallon (12.28 lbs.), $1.05 to $1.10 in large quantities.

This product, it has been stated, has the disadvantage of hydrolyzing sufficiently to cause damage to steel extraction apparatus. The writer has visited numerous European extraction plants which have used trichlorethylene for a number of years, and the managers have assured him that they have not been troubled with corrosion. Edouard Bataille, a manufacturer of extraction machinery, has remarked the same favorable results in the many plants which he has installed in the past thirty years, many of which use this solvent.

In order to determine the comparative action upon different metals, pieces of the various substances were heated over a period of five days in trichlorethylene and water, to observe the effect of the resulting hydrolysis. The results were as follows:

	Per cent loss of weight
Wrought Iron	0.04 %
Cast Iron	0.04 %
Copper	0.03 %
Lead	0.015%

According to this result it would have taken approximately 34 years of constant attack to have corroded the wrought iron in its entirety.

Another series of tests is reported where thin strips of metal were exposed to the action of trichlorethylene and moisture by being placed in a reflux condenser for 10 hours. One set of experiments was made with oil seeds present in the solvent to observe the effect of the presence of free fatty acids and the other with the solvent and moisture alone. The results are reported in loss of weight per 0.5 sq. inch of surface exposed:

	Mild Steel	Wrought Iron	Nickel	Aluminum	Lead
Trichlorethylene with water	Slight	Slight	Nil	Nil	Nil
Trichlorethylene water and oil seeds	0.0025 gm.	0.0028	Slight	Slight	0.0010

Carbon Tetrachloride CCl_4

Boiling Point	170 °F
Freezing Point	-9.4°F
Specific Gravity at 77°	1.583

This non-inflammable solvent has been rejected for use with steel solvent extraction apparatus because of its marked tendency to hydrolyze. It has been recommended that the hydrochloric acid can be neutralized by the addition of a small amount of hydrated lime in the bottom of the still but this is evidently impossible to do in the distillation of the solvent from vegetable oils.

CRUDE OIL ANALYSIS

By DAVID WESSON

In the September issue an account was given of an accurate method for determining exactly the amount of refined oil in any given sample of crude. The application of this method to factory control and the purchasing of crude oil was not mentioned.

Knowing the amount of refined oil in a given sample of crude and the efficiency of factory refining operation it is very evident that a factor can be used to show the maximum loss which should be expected.

It is evident in studying the tables, 5a, 5b, 5c and 5d*, that the average refining efficiency on the prime oils handled was 96.3 per cent, while the average kettle yield was 93.85 per cent of the crude worked. The absolute oil found, multiplied by refining efficiency, should equal the kettle yield.

The average absolute oil from the 40 refinings in the above tables was 97.3 per cent, which multiplied by the average efficiency gives us 93.7 —the average yield to be expected. 100 minus 93.7 equals 6.30 average refining loss.

This can be arrived at another way by adding the absolute loss to 100 minus efficiency per cent. Thus, the average efficiency being 96.3 per cent we have efficiency loss of 3.70. 3.70 plus the average absolute loss of 2.70 gives us a loss of 6.40, as against 6.30 as shown above.

When it is considered that the 40 refinings were all made at a very high efficiency, it would seem fair to add a factor of 4 to the absolute loss on an oil, assuming a refining efficiency of 95. This would give a factor of safety in buying oils, and at the same time be a nearer approximation to average kettle results than we obtain by the hard refining tests as now carried out.

The present refining test does not agree closely with the kettle results in most cases for the following reasons: First, the sample tested in the laboratory does not represent the oil in the kettle unless taken from a kettle during thorough agitation.

Average tank car samples taken from the tank cars are not accurately representative. This is proven by comparing the acidity of such samples with that of the same oil after thoroughly mixing in a refining kettle.

Second, the refining conditions in the laboratory differ greatly from those in the kettle and very different results are frequently obtained.

With off oils, the stirring apparatus in both kettle and the laboratory is very inefficient as can be proven by results. The efficiency can be determined exactly by means of the absolute oil test, and the correction of refining apparatus becomes a mechanical problem.

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*Journal of Oil & Fat Industries, 3, 9, 300-301.

Referring to the previous communication, one or two points should be made clear. Any good 95% alcohol or formula 30 can be used in making the test.

Caustic potash is used instead of caustic soda because it forms a liquid soap easily soluble in the alcohol water mixture.

The quantity of caustic potash used is equal to 10 per cent of caustic soda on the weight of the oil. It is therefore sufficient to saponify 70 per cent fatty acid in the oil and is many times the amount ever used in practice.

In conclusion it is recommended that the value of crude oils be determined by determining the amount of absolute oil contained and estimation of the probable loss by adding a suitable factor to the absoluate loss.

In addition to the above test a laboratory refining should be made with a specified excess of caustic soda of sufficient strength to determine the color of refined oil which can be made from a given sample of crude.

CAN BORROW ON COTTONSEED

Cottonseed warehouse receipts will be regarded as sound and acceptable collateral under regulations just promulgated by the Secretary of Agriculture for storage of such seed, as the result of a careful study of the commodity by department officials and the Federal Reserve Board.

Some time ago the board ruled that cottonseed was non-perishable when stored under proper conditions, and readily marketable within the meaning of the Federal Reserve act. At a recent conference it was disclosed that many warehouses were owned by the same persons who owned the seed. By previous rulings it was necessary for the commodities, to be good collateral, to be leased to parties having no interest in the product in storage. There was a ruling also that the borrower should have no access to the premises in which the cottonseed was stored.

The department's ruling follows:

"Bankers' acceptances secured by cottonseed stored in a warehouse owned by the owner of the cottonseed but leased to an independent public warehouse corporation under bona fide lease, the corporation assuming executive control and management of such warehouse, operating it as a public warehouse, bonded and licensed under the United States Warehouse act, may be eligible for rediscount at a Federal Reserve Bank, although the owners of the cottonseed are permitted access to the seed in storage at proper and reasonable times for the purpose only of inspecting the condition of the seed; provided that on all such occasions the consent of the independent warehouse corporation is first secured, and that the owner of the seed or his representative is accompanied by a proper representative of the warehouse corporation."

THE CHILLING AND PRESSING OF COD LIVER OIL

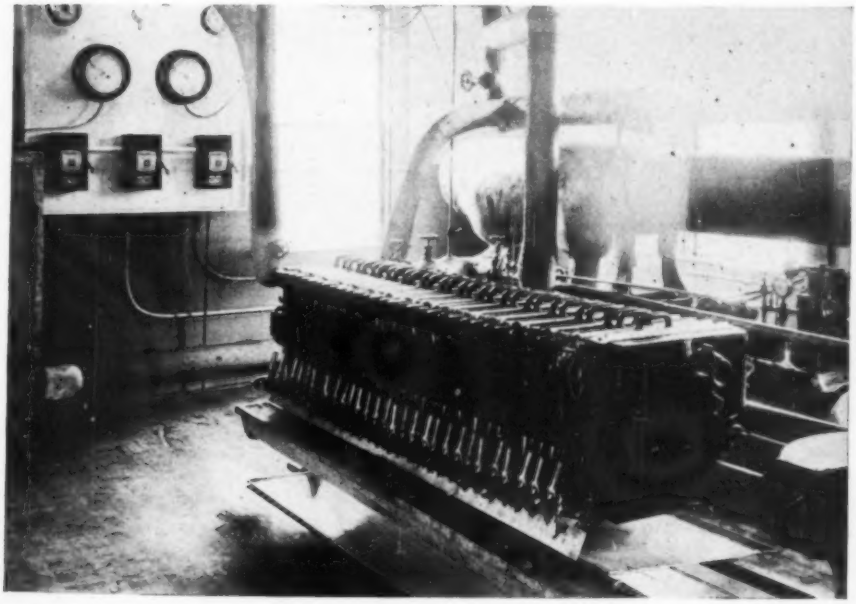
For a long time it has been a universal practice to chill and press crude cod liver oil to remove the cod liver stearin from oil that is intended for medicinal purposes.

Crude cod liver oil when made from strictly fresh livers under modern manufacturing conditions does not differ markedly in color, odor and taste from the pressed oil which is obtained from it by chilling and removing the stearin. Furthermore, our tests show that the vitamin potency of crude oil and the medicinal oil pressed from it is practically the same.

The objection to crude oil for medicinal purposes is that at low temperature the stearin solidifies. The oil becomes cloudy, somewhat thickened, or possibly semi-solid depending on the temperature. Accordingly, to make a more attractive product, manufacturers have for a long time removed the stearin from cod liver oil intended for medicinal use.

In the early days the method for removing stearin was rather crude. The manufacturer pressed "on the weather," by which he meant that he held his oil until winter, when nature supplied the required temperature necessary for solidifying the stearin.

In the early attempts at artificial chilling of cod liver oil, the manufacturer placed an ice and salt mixture around metal cans containing oil. These cans were rectangular, narrow and deep, and very similar to those



Equipment described in the text for chilling and pressing cod liver oil

used in the artificial manufacture of ice. While this procedure served to satisfactorily chill the oil, it was not a complete success because the manufacturer could not press the chilled mass at ordinary temperatures without some of the stearin melting and becoming mixed with the non-freezing oil.

The next step in the development of artificial chilling of cod liver oil was to provide a brine cooled room of sufficient size to contain an open tank for chilling the oil and a press for separating the stearin. While a decided improvement over anything that had been previously used, this process was still open to a number of objections. It was expensive due to the large space that had to be maintained at a low temperature. Two or three days were required to obtain a satisfactory crystallization of stearin. The pressing of the oil was attended with a certain amount of atmospheric oxidation, which destroyed the vitamin potency of the oil to some extent. Obviously, in order to produce the highest type of product, it was necessary to conduct the chilling and pressing process under the best possible conditions. As a consequence, the equipment shown in the accompanying photograph was developed.

At the back of the photograph the reader will notice two horizontal pipes. In the lower pipe the cod liver oil is chilled. It consists of two concentrate pipes, the inner of which contains the oil and a spiral worm for moving it towards the press; the outer insulated pipe contains the low temperature brine. The chilled mass slowly moves into the press where the stearin is removed. From there the non-freezing oil passes to the receiving tanks on the floor below.

Those of our readers who are mechanically inclined will be interested to know that the two dials shown on the center of the instrument board are recording thermometers; one of these gives a constant record of the temperatures of the brine, and the other constantly records the temperature of the oil that is being chilled.

By means of this type of equipment, we rapidly reduce the temperature of the crude cod liver oil to a point where the stearin solidifies. Also the chilling and pressing is all done out of contact with the air.

It is needless to point out that by means of modern equipment of this type, the present day manufacturer can produce a cod liver oil far superior to that manufactured in the earlier days.

—Patchwork. E. L. Patch Co., Boston

THE COMPOSITION OF CRUDE COTTONSEED OIL; A SUMMARY

By GEORGE S. JAMIESON and WALTER F. BAUGHMAN

Cottonseed oil is the most important edible oil produced in the United States and in recent years the percentage of the output used for alimentary purposes has been increasing. The leaders in the cottonseed oil industry realize the importance of a close acquaintance with the raw material, i.e., the character and composition of the crude oil, because this familiarity will help to increase the efficiency of the present processes and aid in developing new ones. Several associations and societies have requested that the composition of the crude oil be studied by the Department of Agriculture. This paper summarizes the results so far obtained from the investigations conducted in the Oil, Fat and Wax Laboratory.

Physical and Chemical Characteristics

In Table 1 are tabulated the variations and averages of the results obtained by the analyses of 41 authentic commercial samples of oil.* The samples were collected at the mills in nine southern states during the pressing season 1919-20 and represent the current production of hot pressed oil. The acidity test was made on the crude oil. For the other determinations, oils refined according to the official methods of the American Oil Chemists Society and the Interstate Cottonseed Crushers' Association were used.

TABLE 1. ANALYSES OF AUTHENTIC AMERICAN COTTONSEED OILS
1919-1920

		Acidity Oleic Acid Per Cent	Refining Loss Per Cent	Titer	Refractive Index at 20° C.	Specific Gravity 25°/25° C.
Average		1.9	8.7	34.4	1.4695	0.9173
Usual Variations	High	3.0	10.8	35.0	1.4700	0.9174
	Low	0.9	5.3	33.8	1.4690	0.9171
Extremes	High	9.2	29.2	35.6	1.4720	0.9181
	Low	0.6	3.6	32.2	1.4768	0.9168

		Iodine No. (Hanus)	Saponification Value	Saturated Acids Corrected Per Cent	Unsaturated Acids Corrected Per Cent	Iodine No. (Hanus) of Unsaturated Acids
Average		107.4	194.7	23.5	71.5	146.3
Usual Variations	High	109.0	195.7	24.2	72.3	147.0
	Low	106.0	193.9	22.9	70.6	145.0
Extremes	High	111.2	195.7	25.3	73.9	148.0
	Low	103.0	193.9	20.9	69.7	144.2

* For detailed results see Cotton Oil Press 4: 3, 85 (July, 1920; and 4: 6, 48 (October, 1920).

Neutral Oil

The most important constituent of crude oil is, of course, the neutral oil. It is possible to determine the amount present by the David Wesson method,* which, briefly, is as follows:

A weighed sample of crude cottonseed oil (about 10 g.) dissolved in petroleum ether is shaken with 10 cc. of a 14 per cent potassium hydroxide aqueous solution. Then 25 cc. of 50 per cent alcohol is added and the agitation repeated. By this treatment the free fatty acids are neutralized and dissolved in the alcoholic alkaline solution, the dark coloring matter is extracted, and very small quantities of certain fatty acid compounds other than fatty acid triglycerides are saponified. The mixture is allowed to stand until it separates into two layers, an upper layer consisting of a petroleum-ether solution of neutral (refined) oil and a lower layer containing the potassium salts of the fatty acids, the coloring matter, and other impurities. The two layers are separated. The solvent is distilled off and the residue, which consists of neutral oil, is weighed. The oil thus obtained has about the same color as that obtained by the official refining loss test. The refining loss (Wesson method) or the percentage of concomitants other than neutral oil in the crude oil is obtained by substracting the percentage of neutral oil from one hundred.

TABLE 2. ANALYSES OF CRUDE COTTONSEED OIL

Sample	Neutral Oil Per Cent	Refining Loss (Wesson Method) Per Cent	Fatty Acids in Wesson Refining Loss Per Cent	Free Fatty Acids (Calculated as Oleic Acid) Per Cent	Refining Loss (Official Method) Per Cent
1	98.07	1.93	1.41	0.65	6.69
2	97.82	2.18	1.99	1.42	12.00
3	96.80	3.20	2.23	1.57	10.40
4	96.28	3.72	2.96	2.05	8.60
5	95.98	4.02	2.98	2.19	8.20
6	94.05	5.95	4.99	3.79	18.56
7	87.42	12.58	11.43	9.18	29.60

To determine the total fatty acids and the small amounts of other acids neutralized by the potassium hydroxide in the alcoholic alkali solution, the alcohol is evaporated, about 100 cc. of water is added, and the fatty acids are precipitated with hydrochloric acid. The mixture is then heated until the fatty acids collect on top of the solution and cooled until they solidify, when a separation is made by filtration. After being washed with water and dried, the acids are dissolved off the filter with petroleum ether and weighed after distilling off the solvent. This determination is entitled "fatty acids in refining loss."

Table 2 gives some results of analyses of crude oil by the foregoing

* Detailed procedure for making this determination and several others was published in The Cotton Oil Press 6: 4, 33 (August, 1922).

methods. For comparison the percentage of free fatty acids and the refining loss by the official method are included.

The Wesson procedure does not decompose fatty acid triglycerides. When applied to dry neutral oils, 100 per cent recovery of neutral oil was obtained and when applied to commercially refined oils a refining loss amounting to 0.20-0.22 per cent resulted. This is equal to the sum of the percentages of moisture (0.14-0.16) and acidity (0.04-0.06).

The official refining loss test, used by the cottonseed oil trade to evaluate crude oil, simulates the commercial refining process (alkali method) and determines more or less satisfactorily the commercial refining loss and how much refined oil can be obtained from the crude. The percentages of refined oil obtained by this test are always considerably less than the percentages of neutral oil determined by the Wesson method, because the emulsion formed at first when refining by the official method does not break as completely with some oils as with others and different quantities of neutral oil are carired down by the "soap stocks." For the same reason the commercial refining loss is always greater than the refining loss determined by the Wesson method. The two do not run parallel and the commercial loss is not proportional to the free fatty acids in the crude. The percentage of neutral oil represents the goal for which the refiner is to strive.

Composition of Neutral Oil

The approximate chemical composition of neutral oil is given in Table 3. These results were determined on an authentic sample of cottonseed oil (J. Am. Chem. Soc. **42**, 1197 (1920). The procedure used was briefly as follows:

The saturated and unsaturated acids were separated by the lead-salt-ether method. The saturated acid fraction was esterified with methyl alcohol. The methyl esters were distilled fractionally under diminished pressure in such a way that the fractions did not contain more than two esters, and the fractions were weighed. The fatty acids were liberated from the various fractions, separated by fractional crystallization, and identified by elementary analyses and melting point determinations. The percentage composition of each fraction was calculated from the mean molecular weight of the fraction as determined and the theoretical molecular weights of the two esters present. From these data the com-

TABLE 3. COMPOSITION OF NEUTRAL (REFINED) COTTONSEED OIL.

		Per Cent
Glycerides of	Myristic acid	0.3
	Palmitic acid	20.0
	Stearic acid	2.0
	Arachidic acid	0.6
	Oleic acid	35.2
	Linolic acid	41.7

position of the main saturated acids fraction was calculated. The bromine addition derivatives of the unsaturated acids were made. No hexabromide, which is insoluble in ether, was found, proving the absence of the linolenic acid. The ether was removed by distillation and the residue dissolved by boiling with petroleum ether. The linolic tetrabromide isomer, which is insoluble in cold petroleum ether, was crystallized out in the ice box and weighed. The petroleum ether solution, which now contained the soluble linolic tetrabromide isomer and the oleic dibromide, was evaporated to dryness and weighed. The bromide content of the residue was determined and the proportion of tetrabromide and dibromide calculated. The percentages of linolic and oleic acids in the main unsaturated acids fraction were calculated from these data.

Palmitin and Di-Palmitin

Some interesting results which have a relation to the composition of the neutral oil were obtained by examining the "settlings" from a five-gallon sample of high-grade crude oil that had been standing in the laboratory for about three years (J. OIL AND FAT IND. **3**, 5, 153 (May 1926)). The acidity of the oil after this length of time was only 2 per cent, as oleic acid, and it was free from rancidity.

As much as possible of the oil was drained from the "settlings," after which the deposit was washed with cold acetone until free from oil. A 240 g. portion of the residue was repeatedly boiled with hot absolute alcohol until all of the alcoholic-soluble constituents had been extracted. The combined alcoholic extracts, amounting to about three liters, were concentrated to about half the original volume and allowed to remain in a cool place for several days. A crystalline almost white precipitate separated. This was removed by filtration, washed with several small quantities of cold alcohol, and then dissolved in one liter of warm alcohol. The yellow solution was decolorized with norite. On cooling, a bulky white mass composed of minute slender prisms was deposited. After filtering, washing, and drying, the melting point was 63.5°-64.0°C. and recrystallization did not change it. The crystals belonged either to the monoclinic or the triclinic system, but it was not possible to determine which. No free fatty acids were present, but after saponification palmitic acid and glycerine were separated and identified. The saponification value of the product was 208.5. The theoretical saponification value for the triglyceride of palmitic acid is 208.6. These data identify the compound as palmitic-triglyceride. The yield of palmitin was 60 g., or 24.8 per cent of the "settlings."

The alcoholic solution from which the palmitin had been recrystallized was concentrated to a volume of about 200 cc. On standing, a small quantity of another glyceride crystallized out. This was identified as

di-palmitin. It crystallized from alcohol in radiating clusters of minute needle-like prisms, which appeared to belong to the orthorhombic system. Its melting point was 70°C. and saponification value 196.7. All of its properties agree with those of the di-palmitin synthesized by Ad. von Grün (B. **38**, 2285 (1905)). The yield of di-palmitin was 2.6 g., or 1.1 per cent of the "settlings."

So far as known this is the first instance in which the pure glycerides, palmitin and di-palmitin, have been isolated from cottonseed oil.

Mixed glycerides, no doubt, are the preponderant constituents of the neutral oil, but this work establishes the presence of a small quantity of palmitin. The presence of the di-palmitin is believed to be due to the hydrolysis of a small quantity of a triglyceride.

Acetyl Value

At this point it may be worth while to consider for a moment the acetyl value, which is variable and amounts in some cases to 18 or 20. The quantity of phytosterol (theoretical acetyl value 131.1) present is less than 1 per cent. Hence this compound is not responsible for more than 1 in the acetyl value. Cottonseed oil as been examined by different investigators for hydroxylated acids, but none has been detected. Also careful search has been made for free glycerine but none found. It is believed that the acetyl value of cottonseed oil is due chiefly to the presence of di- and mono-glycerides, which is caused by the hydrolysis of some of the triglycerides (Lewkowitsch, Chem. Technology and Analysis of Oils, Fats and Waxes, 6th ed., Vol. 1, p. 440). Therefore, if this assumption is correct, low acetyl values should accompany low acid values, and oils with greater acidities should give higher acetyl values. This, however, remains to be investigated.

Free Fatty Acids

Crude oil, even in its fresh state, contains a small but appreciable amount of free fatty acids and the quantity increases upon storage, especially if the oil has not been properly settled or filtered and contains some "foots" (meal). This is due to the action of fat hydrolyzing enzymes which are present in the seed. Fortunately the alkali refining process destroys or removes any enzymes not previously removed from the crude oil by the settling process or by filtration.

It has been considered that some fatty acids are split off from the glycerides more easily than others and that the free fatty acids of cottonseed oil consist only of oleic acid. For commercial trading it is customary to calculate and express the acidity as oleic acid.

The free fatty acids were separated from five samples of crude oil and examined (Cotton Oil Press **7**, 2, 35 (June, 1923)). It is concluded from the data obtained that the free fatty acids of cottonseed oil

are set free by hydrolysis in practically the same proportions as they occur as glycerides in the oil.

Minor Constituents

The crude oil contains a number of other concomitants in small quantities. Some are pigments, others are strong emulsifiers which exert a marked influence on the amount of neutral oil that is carried down by the "soap stock" in the commercial refining process. The average commercial refining loss is about 8 or 9 per cent unless it is an "off" year, and about 25 per cent of this loss consists of neutral oil that has been carried down, entrained in the "soap stock" owing to emulsification. Therefore some of these other constituents, although they occur in small amounts, are important factors in determining the size of the refiner's profits. The quantity of these substances is indicated by the difference between Column 3, refining loss (Wesson method), and Column 5, free fatty acids, in Table 2. Some of these constituents have been identified.

A convenient manner for obtaining a quantity of these constituents for examination is to use the precipitate which usually appears in crude oil that is a few weeks old. Of course, all press cake or meal is removed from the oil before the precipitate separates. This precipitate is at first very bulky but slowly settles out, sometimes as a fine powder, but usually as a thick gelatinous mass having a dark or almost black color. It is, no doubt, caused by the presence of the small quantity of moisture that is commonly present in the crude oil, as it is known that the addition of 2-3 per cent of water to the crude oil, agitating and warming the mixture, will cause a precipitation of these constituents. When this precipitate first appears in commercial crude oil it appears as fine meal, so that the shipper is sometimes unjustly accused of shipping oil contain meal.

This precipitate, a dark gummy mass, is washed with cold acetone until free from oil. This treatment changes it to a bright yellow powder, which for convenience will be referred to as the "acetone insoluble settlings." The acetone washings also are valuable for examination.

An analysis of the "acetone insoluble settlings" gave the following:

	Per Cent
Chlorides	0.00
Silica	0.60
Phosphorus pentoxide	6.14
Sulphates	0.00
Calcium oxide	0.26
Magnesium oxide	1.46
Potassium oxide	1.79
Sodium oxide	0.33
Ferric oxide	trace
Nitrogen	1.71

Test for starch and tannin were negative. Fehling's solution indicated the absence of reducing sugars, but 4.2 per cent of raffinose (R. T. Balch) and 1.74 per cent of pentosans (F. B. La Forge) were found. Arno Viehoever and Ruth Capen could not detect the presence of quercetin, isoquercetin, quercimeritrim, or gossypitem which are present in the cotton plant, but their observations indicated the presence of xanthopyll and a wax. Charles E. F. Gersdorff obtained the following results: Tests for unaltered cottonseed protein, as well as tests for acid and alkaline meta-proteins, were negative. All tests for proteoses and peptones were positive and indicated the presence of a considerable quantity of these substances. There is no known method for making quantitative determinations of them in a mixture of this character.

The "acetone insoluble settlings" is partially soluble in water, ether, chloroform, and absolute alcohol. The aqueous extract is neutral.

Resins

When the alcohol is evaporated from a portion of the alcoholic extract of the settlings a brown residue remains. Boiling this residue with alcoholic potash causes the solution to become dark red and to deposit a crystalline salt of resin acids, which readily dissolves upon adding water. Upon acidifying the solution with hydrochloric acid a characteristic light-brown precipitate of resin is obtained. The resin is soluble in alcohol and produces a strongly reddish-yellow solution. The addition of an alcoholic solution of cadmium chloride gives no precipitate, which shows the absence of phospholipins such as lecithin. An alcoholic solution of copper acetate causes a dark yellow flocculent precipitate to separate. If oil from which the settlings have been removed is agitated with 2-3 per cent of water and heated to about 50°C., more resin, together with other substances, is precipitated. Furthermore, alcohol will extract some resin from a water treated oil. The caustic soda refining process, however, removes practically all of the resin. It occurs in the glands that permeate the kernels, not in the hulls of the cotton seed. Oil expressed from prime seed contains less resin than "off" oil from stored seed that contained excessive moisture and therefore "heated" and deteriorated. The resin was found to have strong emulsifying power.

Inosite Phosphates

Inosite phosphates may be separated from both the "acetone insoluble settlings" and the aqueous extract which is obtained by heating the oil with a small quantity of warm water. They are moderate emulsifiers.

It is interesting to note that the principal phosphorus compounds found in the seed by R. J. Anderson (J. Biol. Chem. **13**, 311 (1912) and **17**, 141 (1914)) and J. B. Rather (J. Am. Chem. Soc. **39**, 777 (1917)) are inosite phosphates.

Phospholipins, Phytosterols and Phytosteroline

Part of the phosphorus occurs in compounds which are related to the vegetable lecithins. The phospholipins may be precipitated in the form of cadmium chloride compounds from methyl alcohol extracts of both the "acetone insoluble settlings" and the oil. They may be precipitated also from a concentrated alcoholic extract by the addition of acetone. Repeated extraction of the crude oil with methyl alcohol removes only a portion of the phospholipins, but the caustic soda refining process removes practically all of the phosphatides. The phospholipins, like lecithin have strong emulsifying powers. Up to the present time no method has been found by which it is possible to determine the quantity of either the phospholipins or the inosite phosphates in the oil or in the "settlings."

A large part of the unsaponifiable matter consists of sterols. R. J. Anderson and M. G. Moore (J. Am. Chem. Soc. **35**, 1944 (1923)), who have made a very thorough study of the phytosterols in cottonseed oil, report the presence of at least two phytosterols which differ in melting points and optical activities. The phytosterols are moderate emulsifiers.

This glucoside, a compound of glucose and phytosterol, has been obtained from the concentrated alcoholic extract of the "acetone insoluble settlings" and purified by repeated crystallization from alcohol. When heated it turns brown at about 250°C. and melts with decomposition at 275°-276°C. It gives an intense reaction for sterol when tested in the usual manner with acetic anhydrid and sulphuric acid. The acetyl derivitive melts at 166°-167°C. An elementary analysis gave the following results: Carbon 72.16 per cent; hydrogen, 10.12 per cent. The theoretical percentages for $C_{27}H_{45}O.C_6H_{11}O_5$ are carbon 72.20 and hydrogen 10.27. It agrees in its properties with the phytosteroline from other sources previously described by F. B. Power and A. H. Salway (J. Chem. Soc., **103**, 399 (1913)). It constitutes about 0.1 per cent of the "settlings" and is not an emulsifying agent. A somewhat similar substance has been isolated from the benzene extract of cotton lint by R. G. Fargher and M. E. Probert (J. Textile Inst., **14**, T 49 (1923)).

Mucilaginous Substances

The alcohol-insoluble portion of the "acetone insoluble settlings" is partially soluble in water and mucilaginous material has been found in this water solution. It has not been possible to make a quantitative determination because when extracting in paper thimbles with boiling water the pores of the thimbles soon become clogged and other procedures are not more satisfactory. This aqueous extract is neutral in reaction. When a 20 per cent solution of normal lead acetate is added in excess, a slightly colored lead compound is precipitated. The lead compound may be suspended in water decomposed with hydrogen sulphide, and the lead sul-

phide removed by filtration. Adding approximately 30 per cent of alcohol to the filtrate gives a white gelatinous precipitate of the mucilage. It is a strong emulsifier, but fortunately for the refiner it is present only in very small quantities.

Basic lead acetate added to the filtrate from the lead compound of the mucilage produces no precipitate, which shows the absence of water-soluble gums in the "settlings."

The Coloring Matter

Many attempts have been made to separate the pigments, such as chlorophyll carotin, etc., from the oil for the purpose of identification, but so far these attempts have failed. Apparently, the only feasible method for the identification of these substances in oils is by means of the spectrophotometer. The spectrophotometric examination of both animal and plant pigments has been in progress for a number of years and some definite results have been obtained. Considerable progress has been made by I. G. Priest (The Cotton Oil Press, **3**, 3, 86 (1919) & **3**, 9, 37 (1920)) and his associates in the use of this instrument for measuring the colors of refined cottonseed and other vegetable oils and some attention has been given to the character of the pigments present in these oils. The evidence already obtained indicates the presence of carotin, xanthopyll, and chlorophyll in cottonseed oil. Part of the dark red color of crude cottonseed oil is due to resin, as already described. The color of a crude oil is noticeably lightened after it has been heated with water which has been shown to remove a portion of the resin. On the other hand, the treatment with water removes little or none of the plant pigments dissolved in the oil.

Conclusion

The qualitative composition of crude cottonseed oil is expressed in the following diagram:

- Crude Cottonseed Oil ...
  - Glycerides of.....
    - Myristic acid
    - Palmitic acid
    - Stearic acid
    - Arachidic acid
    - Oleic acid
    - Linolic acid
  - Free Fatty Acids. — Same proportions as in glycerides
  - Minor Constituents
    - Proteoses
    - Peptones
    - Phytosterols
    - Phytosteroline
    - Inosite phosphates
    - Phospholipins
    - Resins
    - Mucilaginous substances
    - Carbohydrates
      - Raffinose
      - Pentosans
    - Pigments
      - Xanthophyll
      - Carotin
      - Chlorophyll

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NEW POSSIBILITIES IN CALIFORNIA OLIVE OIL

By P. F. NICHOLS

The purpose of the present work on olive oil being conducted at the University of California is to render the industry more profitable to manufacturer and grower by increasing the yield of oil or by improving its quality. The work falls in three groups which are, in the inverse order of their probable economic significance, pomace utilization, oil refining and oil standardization.

Pomace Utilization Possibilities

The present system of oil manufacture has one weakness in the fact that the pomace from the presses is either burned directly or thrown away. Since there is usually more pomace produced at a plant than can be used for fuel, there is a labor cost involved in the disposal of the excess. It would seem that such labor might well be engaged in the production of a by-product if one can be found which will be profitable.

Chemical analyses of the pomace, from various sources, are as follows:

	Brannt[1]	Hefter[2]	Cruess and Christie[3]	Du Vauchelle[4]	Nichols[5]
Water	13.20	10.7	—	13.0	25.4 —26.5
Fat	13.20	13.8	7.89—20.23	8.0	10.27—14.4
Ash	6.80	6.5	Ave. (18) 12.41	6.3	—
Non-nitrogenous matter including fiber	60.20	—	—	—	—
Fiber	—	33.7	—	22.1	—
N-free extract	—	28.1	—	43.7	—
Protein	6.00	7.2	—	6.9	—
Nitrogen	—	—	— 0.86	—	—

The tonnage of pomace produced in California in 1925-26 may be estimated on the basis of a total production of 300,000 gallons of oil from olives yielding on the average 40 gallons of oil and 1,200 pounds of pomace per fresh ton. This would result in a production of 4,500 tons of pomace.

There appear to be two principal possibilities for the use of this material: first, as a stock feed, and, second, as a source of further oil. According to Cruess ([6]), olive pomace is a fair stock feed. DuVauchelle ([4]) reports its successful use as an ingredient in horse rations. Professor W. M. Regan of the Animal Husbandry Division at the University Farm at Davis is interested in the possibility of using it as a cattle feed and believes that if it proves sufficiently palatable it may have a value of approximately $25 per ton when dried and ground. As soon as fresh pomace is again available it will be possible to ascertain its value by actual test.

From the analyses given above, it is assumed that a moisture content of approximately 25 per cent and an oil content of approximately 12 per cent fairly represents the pomace produced in the State as a whole. Under these conditions it is believed that it should be a profitable source of oil. This oil might be obtained either by the use of greater pressure than is now

used, or by extraction with solvents. A part of the oil could probably be recovered most economically by the use of greater pressure, either in a continuous screw press of the expeller type, or in a heavy hydraulic press. It is doubtful, however, if more than half the oil could be recovered in this way, reducing the oil content to 5 or 6 per cent as a minimum.

Anything like complete recovery of the oil appears possible only by the use of a solvent extraction process. When it is noted that the ordinary hydraulic pressing yields only about 68 per cent of the oil actually in the olives, it will be seen that the volume of oil added by complete extraction of the pomace would be large, amounting to 140,000 gallons for the whole State in 1925-26. This, when considered for use only as soap oil, would have a value of over $90,000 on the present market, and if it could be successfully refined for food it would have at least twice this value.

Experiments conducted at the Fruit Products Laboratory of the University of California this year indicate that it is easily possible to extract in this way at least 75 per cent of the oil contained in the pomace. In order to do so, however, it was found necessary to dry the pomace. In the case of pomace which had been dried to under 10 per cent moisture, yields of oil were obtained which ranged from 8.5 to 14.0 per cent of the weight of the pomace, using several different solvents, while pomace which still contained about 25 per cent moisture as when it came from the press yielded only about 5 per cent. The oil so obtained was of perfectly satisfactory quality for soap oil, and while this has not yet been accomplished in California, it would appear possible to refine it for use as food, and that claim is made by several manufacturers of refining equipment.

There are several systems in use for the extraction of oil from such materials as olive pomace. One is similar to the Soxhlet apparatus used in the laboratory for the determination of oil content. In this the solvent is kept constantly boiling, the condensed solvent flowing upon the pomace, dissolving the oil and siphoning off at intervals, and the oil recovered by boiling off the solvent after extraction is complete. Another system works upon the principle of a diffusion battery such as used in the production of sugar from sugar beets. In this the oil bearing material is contained in several tanks through which the solvent is run, flowing from the spent through the fresh material, and thus becoming constantly enriched with oil without the necessity of intermittently drawing it off and distilling the solvent. The oil is finally recovered by distillation of the solvent, as described above. Still another type is employed which is similar to the preceding systems, except that the solvent and pomace are kept in motion by agitators or by the rotation of the drums in which the process is conducted.

In the event that extraction plants were to be operated in California,

it would appear that gasoline, or a similar petroleum solvent, could be used to best advantage since such solvents would be considerably cheaper than the others that are available. Some solvent is inevitably lost in the course of the operation, and a cheap solvent would reduce the expense resulting from such a loss, as well as reducing the cost of the initial supply.

It is believed that the cost of such a process, including the overhead and operating costs of drying and extracting the material, could be kept below $8 to $10 per ton of pomace under normal operating conditions with a plant handling 1,000 tons per season. If the yield of oil were 10 per cent, leaving 2 per cent in the pomace, the gross return would be at least $17 per ton. The extracted pomace, when sufficiently dried, would be nearly if not quite as good for use as fuel as it is under present conditions with its high moisture content. This, then, should be a profitable means of utilizing the pomace under conditions as they exist at present.

Were the oil content of the pomace to be lowered to approximately 8 per cent by the use of more powerful presses, solvent extraction might become unprofitable, since the yield of oil would barely cover the cost of the process. It has been suggested that such further pressing might so reduce the moisture content as to render drying unnecessary, thus reducing the cost of the extraction process. While this may be possible, it has not yet been demonstrated.

Olive Oil Refining

In the refining of edible oils such as olive oil, there are usually three steps involved: neutralization of the free fatty acids, decolorizing and deodorizing.

In the neutralization process, an alkaline solution is added to the heated oil in the proper amount calculated from the free fatty acid content. For this purpose caustic soda is generally used commercially, though Cruess (6) prefers sodium carbonate which is cheaper and is less likely to saponify the oil itself rather than the free fatty acids. Experiments conducted at the Fruit Products Laboratory this year seemed to indicate slightly better separation of the soap when sodium carbonate was used under atmospheric pressure, although some of the soap always rose to the top in addition to that sinking to the bottom. Manufacturers of vacuum refining equipment claim a better separation of the soap as a result of the removal of the carbon dioxide which is produced when sodium carbonate is used or is present as an impurity in the lye, tending to become imbedded in and buoy up the soap. In any event, the neutralization of the free fatty acids, to the extent required in olive oil, is not usually troublesome when the carbonate or lye is added in a solution of such strength (12—17° Baumé) as to avoid the formation of emulsions with resultant difficulty in filtration. It is usually the case, however, that a slight excess of alkali must be added in order to secure the desired degree of neutralization.

The decolorization of the oil is also fairly simple when absorptive earths or decolorizing carbons are employed, at least in the oils which have so far been treated. In fact, the refined oils usually retain less color than is desirable, so that they must be blended with more highly colored oils.

The removal of objectionable odors and flavors, whose presence constitutes true rancidity, has proved much less simple and less certain of success, especially when using methods which could be used in the ordinary oil factory without special refining equipment. The neutralization and decolorizing processes, while they have some deodorizing effect, are not sufficient in the case of an oil which is badly off in flavor. Blowing air through oil at room temperature had but slight effect, and while air blown through heated oil had a marked decolorizing effect, it did not improve the flavor. A stream of carbon dioxide through heated oil was also of little if any value in this respect. Treatment of the oil with a current of steam under atmospheric pressure had considerable deodorizing as well as decolorizing effect, and the results seem fairly stable. By all means the most effective method is treating the oil with steam under a vacuum. Oil treated in this way was almost completely decolorized and deodorized. When this method is used commercially, superheated steam is employed, but as no means were at hand for superheating the steam at the laboratory, this was not tried. It is probable that no very definite conclusions should be drawn from this work until the oil has been observed through extended storage.

Olive Oil Standardization

It has been said that the greatest need of California olive oil producers is a system of standardization. With the continuance of the splendid co-operation given the University, it would seem that a suitable plan can be developed. Such a plan should include tests and standards for free fatty acids, color, odor and flavor, and should make it possible to produce with perfect regularity oil which will at all times conform to certain specifications from which the element of human judgment should be as far as possible eliminated.

The amount of free fatty acid, color, odor and flavor should be controlled wherever possible by care in handling the olives and the oil. This failing, resort must be had to the suitable refining processes and blending, though it is probably true that refined oils do not have the stability of those which are of satisfactory character without being so treated.

The free fatty acid content is easily and accurately determined by standard chemical methods, and if necessary can be controlled by neutralization. A standard for this characteristic could be readily established.

The color of some edible oils is customarily determined and expressed in terms of an instrument and set of standard color glasses, as the Lovibond tintometer. Such an instrument, with the color glasses needed for the range of olive oils would cost fifty to seventy-five dollars. Preliminary

experiments with solutions of stable inorganic chemicals indicate that potassium bichromate and copper sulphate solutions of proper strength can be so used as to match the colors of olive oils and if this proves successful, it would permit control of color with very inexpensive equipment, and the expression of color standards indirectly in the Lovibond tintometer scale.

The question of odor and flavor, with respect to standardization, is again difficult and an elusive one. At present it is to such a large extent a matter of purely personal judgment that it can hardly fail to present innumerable occasions of dispute and dissatisfaction resulting from differences of opinion, no matter how honest and sincere the parties to the transaction may be. It is hoped that some chemical or physical method may be utilized or developed which will give results so closely correlated with odor and flavor that a standard may be inaugurated which will completely remove this problem from the realm of human judgment alone. At present no such test can be offered. Numerous tests for this purpose are under consideration and will receive careful study. In any event it is probable that rather extended research on this point will be required.

Along with the questions of the refining and standardization of olive oils, it would perhaps be worthwhile to suggest the possibility of establishing, at the proper time, a central plant for the purpose of handling the refining and blending which would be a necessary outgrowth of the establishment of a system of standardization. It would seem probable that such processes could be handled most efficiently under one roof, under the control of the Association, as a whole, and most readily financed and operated on the basis of the volume of oil handled for each member. Such a plan might be of considerable value in marketing as well as in production.

Summary and Conclusions

I. There is a field for increasing the return on oil olives through feeding the pomace to livestock, and through increasing the oil yield by the use of more powerful presses, or exhausting the oil content of the pomace by solvent extraction.

II. Neutralization of free fatty acids and decolorization are refining methods that can be utilized at any plant, but deodorizing is a problem that will probably require special equipment.

III. Work leading to the establishment of olive oil standards is suggested.

IV. In the event of the establishment of standards, a central refining and blending plant is suggested.

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THE THEORY OF THE PRACTICE OF STEAM DEODORIZATION OF SAPONIFIABLE OILS

A Review

BY DAVID WESSON

This is a subject which has been guarded carefully for the last thirty years as more or less of a trade secret. Little has been known of the theory; or if so, it has not been published. Still less has been published about details of apparatus and the practical operation of same.

A very full treatment of the subject largely from a mathematical standpoint was published in the *Journal of the Society of Chemical Industry* for March 26, 1926, by W. Brash. The same author has a second article on the same subject in the same Journal, September 24, 1926, entitled "Application of the Counter Current Principle to the Steam Deodorization of Saponifiable Oils." The reader is advised to read the original article, only the salient points of which are here given.

The method is most generally used for the deodorization of saponifiable oils for edible use in steam distillation under reduced pressure.

It would appear that the odor and taste are both due to the same chemical substances in the oil and it is found invariably in practice that the odor is first removed then the taste. Steam distillation removes these substances. In some cases the constituents have been identified. They appear to consist largely of Ketone.

In rape oil and plants belonging to the cruciferal the odorous bodies are sulfur compounds; in the fish oils, nitrogenous.

The object of the first paper is to examine the process and indicate needed requirements for an efficiently designed apparatus. Its treatments of the stationary or unit deodorizer are distinct from the continuous.

The amount of odoriferous matter present in different oils varies. It is undoubtedly low, 0.1 to 0.5 per cent. Steam functions in the process merely as an inert gas and has no action on the volatile matter.

Summary

The pressure of steam entering the deodorizer is approximately half the pressure of the steam supply.

The relative dimension of the apparatus and form of the outlet may be calculated from the equation given in the paper.

To shorten the time of deodorization the most important factors are low pressure in the deodorizer and high temperature of the oil.

In the application of the counter current principle which amounts to the same thing as continuous deodorization the apparatus used is a tower in which the air is admitted at the top and the steam at the bottom. The whole apparatus is maintained under reduced pressure and the oil

is withdrawn from the apparatus in a finished condition, while the odoriferous matter is carried over with the steam into a condenser at the top of the apparatus.

The advantages which may be claimed are short time of contact with the steam and economy in the use of steam.

OILS AND FATS IN ANCIENT EGYPT

The following was taken from *The Analyst*, September, 1926, and will be of interest to all our readers. It consists of an extract from a paper by A. Lucas on "Problems in Connection with Ancient Egyptian Materials." The extract reads:

"Although fatty matter has frequently been found in Egyptian tombs, in jars in tombs, sooner or later decompose, and as some of the bodies clusive. This is almost inevitable, as oils and fats, unless kept under special air-tight and sterile conditions, which is not the case when placed in jars in tombs, soonor or later decompose, and as some of the bodies formed escape, either by evaporation or by soaking into the material of the containing vessel, all that the analyst has for examination is merely a portion of the products of decomposition. What is left consists of a mixture of fatty acids, and it is only by the separation, purification and identification of these and by a determination of the proportion in which each occurs in the mixture that the nature of the original oil or fat can be known, and, since what remains is generally a portion of that formed and not necessarily a representative portion, the problem may often be insoluble.

"The fatty matter often smells like rancid coconut oil or like rancid butter and, occasionally, of valeric acid, and the fatty acids identifiable are generally palmitic and stearic acids and, less frequently, oleic acid, and in one instance calcium butyrate was found. When the fatty acid consists essentially of stearic acid alone it seems justifiable to assume that the original material has been castor oil, which was well known to the ancient Egyptian, and which is one of the few oils or fats that contain little or no palmitic acid. When the fatty acids consist of stearic and palmitic acids in almost equal proportions it is possible that the original material was an animal fat, such as ox fat. There is, however, as will be realized, considerable scope for further chemical work on the subject."

PRODUCTION AND CONSUMPTION OF OILS

A valuable statistical bulletin covering Animal and Vegetable Fats and Oils has just been published by the Bureau of the Census. The bulletin contains statistics relating to factory production, factory consumption and factory warehouse stocks for the years 1924 and 1925. Three general classes of factories were canvassed to secure the data: (1) The producers of animal and vegetable fats and oils, including expressers, extractors, renderers, and reclaimers; (2) the consumers of fats and oils in the production of other fat and oil products, or products of which fats or oils form a constituent part, embracing primarily refiners, mixers, compounders and the like, and secondarily manufacturers of varnish, soap, oilcloth, linoleum, etc.; and (3) manufacturers of tin plate, textiles, and leather, which do not themselves contain or retain any considerable portion of the fats and oils used as agents in their manufacture.

The figures of the report are too many and too detailed to be reproduced here; but readers can secure the full survey for 5 cents a copy by writing to the Superintendent of Documents, Government Printing Office, Washington, D. C.

Concerning vegetable oils, edible animal fats and fish oils, the following brief statements will be of interest:

Vegetable Oils

The total supply of vegetable oils in the United States, made up of factory and warehouse stocks at the beginning of the year and production and imports during the year, no account being taken of the stocks in the possession of householders and certain classes of tradesmen and dealers, aggregated 3,984 millions of pounds in 1925, as compared with 3,372 million in 1924. The amount exported plus the amount carried over at the end of the year was 727 million in 1925 and 692 million in 1924, leaving for a potential consumption 3,257 million in 1925 and 2,680 million in 1924. During these years the net factory consumption, arrived at by deducting the total of refined oils produced from the total consumption of all kinds was 2,491 million in 1925 and 1,951 million in 1924, indicating an actual movement toward home and local trade consumption of 766 million pounds in 1925 and 729 million in 1924. Of these amounts, linseed oil, edible olive oil, and the refined cottonseed and corn oils constitute almost the total.

Edible Animal Fats

The total supply of edible animal fats, exclusive of oleo stock, for which no data were collected, and not counting the production or stocks of householders, local butchers, meat markets, and wholesale and retail dealers, was 1,670 millions of pounds in 1925 and 2,107 million in 1924.

The total of exports during the year and stocks carried over at the end of the year was 757 million in 1925 and 1,038 million in 1924. The factory consumption in the manufacture principally of lard and butter substitutes for the two years was 79 million and 85 million, respectively, indicating a domestic trade absorption of 834 million in 1925 and 984 million in 1924. Against this decrease of 150 million pounds in the household consumption of edible animal fats, an increase of 321 million pounds in the analysis of the supply and distribution of the commodities falling under that classification.

Fish Oils

The total supply of fish oils for the year 1925 appears to be 233 million pounds compared with 180 million for 1924, the increase of 53 million pounds being made up of 2 million in stocks carried over, 32 million in production and 19 million in imports. There was an increase of 38 million pounds in the consumption of those manufacturing establishments covered by the scope of this inquiry and of 11 million pounds in factory and warehouse stocks. The movement toward other consumption was 19 million pounds in 1925 and 15 million in 1924. This other consumption is made up principally of refined cod-liver, menhaden, and sardine oils, the first for medicinal purposes and the last two by the paint trade for mixing and thinning purposes.

WHAT IS CASTILE SOAP?

A lawsuit of special interest to all of our readers is pending in the complaint filed by the Federal Trade Commission against James S. Kirk & Company. The issue concerns the term "Castile" as applied to soap.

At the request of the JOURNAL OF OIL AND FAT INDUSTRIES, James S. Kirk & Company has prepared the following statement, in which the facts of the case are clearly set forth. The statement reads:

"On February 29, 1924, James S. Kirk & Company was singled out among other manufacturers of Castile Soap in this country and named as respondent in a complaint filed against it by the Federal Trade Commission of the United States. The gist of the complaint was that James S. Kirk & Company in designating certain brands of soap, which did not contain olive oil to the exclusion of other fatty and oil ingredients, as Castile, deceived the public and thus was unfair in its competition with those manufacturers and importers of Castile Soap made exclusively of olive oil.

"The Commission asserted that Castile as a designation for soap signified solely an olive oil ingredient.

"James S. Kirk & Company for a period of 84 years has been continuously engaged in the manufacture of soap in the City of Chicago. Its business career has been most honorable and its reputation unimpeachable. For over one-half a century American manufacturers of soap have made and sold to the American public vast quantities of soap labeled Castile which did not contain olive oil as an ingredient thereof. The volume of production of American soap manufacturers today of non-olive oil Castile Soap is very large. The volume of imported olive oil Castile Soap is minute in comparison.

"It is most properly contended by James S. Kirk & Company that the name Castile when applied to soap, because of its usage by the trade and by the public, has no significance as to an olive oil ingredient. It seems undeniable that the name Castile means to the trade and to the public a pure high-grade toilet soap. The respondent is joined by practically all American soap manufacturers in its belief that Castile does not mean to the trade or to the public a soap of which the sole oil ingredient is olive oil.

"The motion of James S. Kirk & Company to dismiss the complaint was denied by the Federal Trade Commission. A hearing was held in Washington on October 4th, 1926, at which date the Commission began the introduction of its evidence. The respondent intends to vindicate the honesty of its branding and to protect for the soap trade and the American public the present meaning and usage of the word Castile. Testimony is being secured from all branches of the soap trade and from the public."

CENSUS OF OLEOMARGARINE MANUFACTURES

The Department of Commerce announces that according to data collected at the biennial census of manufactures, 1925, the establishments engaged primarily in the manufacture of oleomargarine in that year reported a total output valued at $39,856,000, an increase of 10.9 per cent as compared with $35,953,000 in 1923, the last preceding census year.

The total production of oleomargarine in the United States in 1925 was 236,495,000 pounds valued at $47,675,000, of which 170,233,000 pounds valued at $34,680,000 were made in establishments engaged primarily in the manufacture of this product and 66,262,000 pounds valued at $12,995,000 were made by establishments engaged primarily in slaughtering and meat packing.

Of the 38 establishments in the oleomargarine industry reporting for 1925, 7 were located in Illinois, 4 in California, 4 in Ohio, 4 in Rhode Island, 3 in Maryland, 2 in Missouri, 2 in New Jersey, 2 in Wisconsin, and the remainder in 10 other states.

Summary statistics for the industry are given in Table 1, below; and statistics of products, by kind, quantity and value, are presented in Table 2. The figures for 1925 are preliminary and subject to such correction as may be found necessary upon further examination of the returns.

Table 1. Summary for the United States: 1925 and 1923

	1925	1923	Per cent of increase
Number of establishments	38	31	...
Wage earners (average number)[a]	1,639	1,484	10.4
Maximum month	Dec. 1,863	Oct. 1,657	...
Minimum month	July 1,552	July 1,302	...
Per cent of maximum	83.1	78.6	...
Wages	$2,278,000	$1,921,000	18.6
Cost of materials (including fuel, electric power, and containers	$28,384,000	$25,772,000	10.1
Products, total value	$39,856,000	$35,953,000	10.9
Value added by manufacture[b]	$11,472,000	$10,181,000	12.7
Horsepower	6,740	5,922	13.8

a Not including salaried employees.
b Value of products less cost of materials.

Table 2. Oleomargarine production: 1925

	Pounds	Value
Total	236,495,000	$47,675,000
Made in oleomargarine establishments, total	170,233,000	34,680,000
Composed of a mixture of animal and vegetable oils and fats	79,923,000	17,085,000
Composed solely of vegetable oils	90,310,000	17,595,000
Made in slaughtering and meat-packing establishments[a]	66,262,000	12,995,000

a Separate data for products of animal and vegetable origin and of vegetable origin exclusively were not reported.

Domestic Exports of Lard Compounds and Oleomargarine During August

Countries	Lard compounds containing animal fats		Oleomargarine of animal or vegetable fats		Vegetable oil lard compounds	
	Pounds	Dollars	Pounds	Dollars	Pounds	Dollars
Belgium	....	....	....	....	480	108
Soviet Russia in Europe	....	....	....	....	2,016	369
Sweden	....	....	....	....	250	44
United Kingdom	2,800	557	....	....	119,942	18,275
Canada	3,562	623	....	....	10,862	1,576
British Honduras	1,991	319	50	12	7,721	1,272
Costa Rica	....	....	224	51	300	55
Guatemala	1,908	363	....	....	9,787	1,781
Honduras	688	134	....	....	3,472	691
Nicaragua	5,890	949	....	....	1,110	187
Panama	10,090	1,526	20,080	3,447	1,607	398
Mexico	29,295	4,574	330	76	120,082	18,541
Newfdld. & Labrador	625	106	....	....	2,500	450
Bermuda	620	102	360	71	2,712	667
Jamaica	868	144	....	....	8,831	1,850
Trinidad & Tobago	600	90	....	....	1,260	275
Other Brit. W. Indies	10,600	1,817	12,575	2,291	11,476	1,922
Cuba	54,414	6,375	200	40	70,373	11,468
Dom. Republic	....	....	570	106	....	....
Dutch W. Indies	10,200	1,614	....	....	1,025	180
French W. Indies	750	122	....	....	....	....
Haitian Republic	22,098	4,238	....	....	79,460	18,698
Virgin Is. of U. S.	15,903	2,413	3,885	753	3,064	485
Argentina	....	....	....	....	10,000	1,700
Chile	7,400	1,258	....	....	5,688	972
Colombia	....	....	....	....	144	31
Peru	....	....	....	....	72	15
Uruguay	....	....	....	....	2,200	407
Venezuela	....	....	....	....	1,404	307
British India	....	....	....	....	90	25
China	2,988	691	100	17	6,270	1,431
Japan, including Chosen	....	....	....	....	6,004	1,318
Kwangtung, 1. terr.	....	....	....	....	94	11
Philippine Islands	....	....	750	225	4,885	1,180
British Oceania	....	....	....	....	72	18
French Oceania	963	141	....	....	867	188
New Zealand	....	....	....	....	7,000	1,190
British S. Africa	224	38	....	....	....	....
British W. Africa	2,000	300	....	....	....	....
Total	186,477	28,494	39,124	7,089	503,120	88,085

Shipments from the United States to non-contiguous territories.

Lard compounds containing animal fats

Alaska	23,156	4,563
Hawaii	78,849	13,189
Porto Rico	43,750	6,163

Domestic Exports of Oleo Oil, Tallow, Oleic Acid or Red Oil, and Other Animal Greases, Oils and Fats, N. E. S., During August

Countries	Oleo oil Pounds	Tallow Pounds	Oleic acid or red oil Pounds	Other animal greases, oils and fats, n.e.s. Pounds
Belgium	53,486	.....	.....	1,257,024
Denmark & Faroe Islands	354,781	.....	.....	.....
Finland	38,228	.....	.....	.....
Germany	1,828,219	343	.....	3,041
Greece	209,032	.....	.....	.....
Irish Free State	58,254	.....	.....	.....
Italy	.....	.....	.....	18,174
Netherlands	1,257,190	41,533	.....	2,820,835
Norway	397,337	.....	.....	.....
Spain	.....	.....	.....	355
Sweden	114,761	.....	.....	.....
United Kingdom	1,828,502	509,324	42,872	1,190
Canada	11,100	239,245	23,923	1,044,602
British Honduras	.....	3,942	.....	.....
Costa Rica	.....	300	.....	31,997
Guatemala	.....	51,005	.....	218,677
Honduras	.....	.....	.....	24,047
Nicaragua	.....	10,000	.....	61,989
Salvador	.....	.....	.....	45,579
Mexico	4,515	199,416	38,116	577,002
Newfoundland & Labrador	238,617	.....	.....	.....
Jamaica	.....	200	.....	.....
Trinidad & Tobago	.....	250	.....	.....
Other British W. Indies	.....	911	.....	.....
Cuba	72,192	71,177	3,964	987,414
Dom. Republic	.....	39,612	.....	.....
Dutch W. Indies	.....	400	.....	.....
Haitian Republic	.....	600	.....	.....
Virgin Is. of U. S.	.....	366	.....	.....
Colombia	.....	81,343	.....	20,200
British India	.....	.....	11,250	.....
Hongkong	.....	4,000	.....	.....
Japan, including Chosen	11,700	.....	.....	4,132
Philippine Islands	.....	.....	.....	15,575
Australia	.....	.....	139,071	1,991
New Zealand	.....	.....	.....	1,188
Egypt	.....	.....	.....	1,948
Total Quantity	6,477,914	1,253,967	259,196	7,136,960
Total Value	$870,105	$117,372	$18,844	$696.029

Exports of Vegetable Oils, Expressed, from the United States During August

Countries	(Cottonseed Oil) Crude Pounds	(Cottonseed Oil) Refined Pounds	Corn oil Pounds	Cocoa butter Pounds	Coconut oil Pounds	Linseed oil Pounds	Soybean oil Pounds
Belgium	.....	92	....	....	.....	.....	....
Germany	22,284	.....	....	....	.....	.....	....
Norway	.....	8	....	....	.....	.....	....
Sov. Russ. in E.	.....	.....	....	....	.....	2,000	....
United Kingdom	.....	.....	750	....	.....	668,330	....
Canada	48,940	19,602	1,888	1,175	542,303	30,595	1,469
British Honduras	.....	182	80	....	401	.....	....
Costa Rica	.....	40	2,400	....	12,520	4,500	12,545
Guatemala	.....	1,038	3,000	24	22,353	4,550	3,750
Honduras	.....	1,142	....	....	.....	5,785	....
Nicaragua	.....	365	....	....	50,074	3,403	750
Panama	.....	750	....	144	1,682	1,163	....
Salvador	.....	2,362	....	....	2,010	3,100	....
Mexico	61,800	49,689	1,134	231	561,706	27,213	....
Newfld. & Lab.	.....	10,000	....	....	230	81	....
Bermuda	.....	1,494	80	....	.....	.....	....
Jamaica	.....	1,175	....	....	.....	.....	....
Trin. & Tob.	.....	100	....	....	.....	.....	....
Other Br. W. I.	.....	1,445	....	....	50	350	....
Cuba	....	4,975	16,875	628	5,651	80,907	63,375
Dom. Repub.	.....	2,652	2,262	....	.....	2,613	42,015
Dutch W. Ind.	.....	2,000	1,195	....	.....	.....	....
Haitian Repub.	.....	6,340	2,625	....	.....	1,786	....
Virgin Is. of U. S.	.....	275	1,450	....	.....	138	585
Bolivia	.....	77	....	....	.....	.....	....
Brazil	.....	.....	....	....	.....	1,611	....
Chile	.....	.....	....	....	4,815	8,300	16,000
Colombia	.....	14,304	5,700	516	5,059	7,590	....
Ecuador	.....	632	....	....	.....	.....	....
Dutch Guiana	.....	2,100	....	....	.....	.....	....
Peru	.....	210	....	385	.....	3,400	....
Venezuela	.....	1,702	....	....	.....	2,251	....
Ceylon	.....	90	....	....	.....	.....	....
China	.....	12,852	....	....	810	.....	....
Hongkong	.....	956	....	....	.....	.....	....
Japan, including Chosen	.....	18,653	....	....	46,825	.....	....
Philippine Islands	.....	9,330	620	....	.....	.....	....
Siam	.....	168	....	....	.....	.....	....
Australia	.....	.....	....	....	.....	9,850	....
French Oceania	.....	90	140	....	.....	3,145	....
New Zealand	.....	.....	....	....	9,100	.....	2,000
British South Africa	.....	2,180	8,100	....	.....	.....	42,120
Port. E. Africa	.....	.....	1,800	....	.....	.....	4,500
Total—Quantities	133,024	169,070	50,099	3,103	1,266,089	812,661	189,109
Total Values	8,836	30,501	7,163	1,196	120,870	31,750	23,656

Shipments from the United States to Non-Contiguous Territories

	Oils, Expressed			
	Cottonseed		Linseed	
	Pounds	Dollars	Pounds	Dollars
Alaska	7,690	1,352	5,049	744
Hawaii	19,060	3,699	56,712	10,900
Porto Rico	6,471	1,107	27,616	3,519

ABSTRACTS OF ADDRESSES BEFORE THE AMERICAN CHEMICAL SOCIETY, PHILADELPHIA, SEPT. 6-10

Phenomena to Be Considered in the Storage of China Wood Oil. Francis W Hopkins. A large amount of China wood oil received in December, 1925, was found to have turned to a semisolid.

Refer to Scientific Circular 270 of the Paint and Varnish Manufacturers Association, May, 1926, the last two paragraphs. Oil was heated and drawn in tank wagon to storage tanks. The following is an analysis of the oil when received:

Color	Brown
Odor	Characteristic
Appearance	Clear
Sp. Gr. 15° C.	0.9410
R. I. 25° C.	1.5185
Acid	4.86
Brown's heat test	11½ minutes

Two months later it was noticed that a spongy-like polymer had formed at the top of the tank to a depth of six to eight inches, the oil at the bottom of the tank was a semiliquid, and an analysis of the oil drawn from the bottom and heated is as follows:

Color	Brown-yellow
Odor	Characteristic
Appearance	Cloudy, opaque
Sp. Gr. 15.5° C.	0.9414
R. I. 25° C.	1.5127
Brown's heat test	9½ minutes
Acid	6.20

Samples from the top of the tank and from the bottom gate were sent to one of the leading experts on China wood oil in the paint and varnish industry, and the analysis is as follows:

Sample from top of tank 45 per cent oxidation products, of alpha and beta calostearin; 55 per cent of alpha calcostearin and olein. Sample from bottom gate 87.5 alpha calcostearin and olein 12.5 per cent beta calcostearin.

After getting in touch with several large varnish manufacturers we drew the following conclusions from their responses, and from a large amount of data which we had obtained.

1. That there was a catalyzer present in the tanker, such as sulfur and the tanker not being quite full, there was a sufficient amount of air present to oxidize the top and the calcostearin formed at the top was insoluble in the oil at the bottom. We found that by skimming this oxidized product in 24 hours about two or three more inches formed, which appears like a reproducing action in the presence of air.

2. It has been found that when a tung nut of high oil content has been pressed at a very high pressure, the oil after standing a year in a closed can formed a considerable amount of beta calcostearin, and which there was a small amount of the oil poured from the can, so that there was an air space at the top, about a month later the oxidized products formed, such as the polymer at the top of our tanks.

3. That this oil was struck by the sunlight when it was in its original baskets in China, and after being in a tank for several months, which is heated, the formation of the beta calcostearin was increased and that in the presence of air in the tanker; the oxidation products of the two acids started a formation on top.

Not only has oil of this nature been received by the paint and varnish industry, but by other industries which use China wood oil. At the writer's suggestion, a large manufacturing concern which had been experiencing trouble with its China wood oil tested for sulfur, of which a small trace was found. Previous shipments were also examined, but no sulfur could be found. The spongy-like oxidized products of alpha and beta calcostearin were removed from the tops of our storage tanks, and the remaining semiliquid heated to a temperature of 300° F., and drawn from the tanks. This oil can be used very satisfactorily in the manufacture of varnish by the addition of a small amount of drier other than that which the formula calls for.

Conclusion: A test of the oil, or series of tests, should be compiled by the

various societies interested in the paint and varnish industry so that such a condition could be detected when the general analysis is made of a sample.

Studies in the Boiling of Linseed Oil. J. S. Long, C. J. Knauss and J. G. Smull. Linseed oil was heated at 293° C. in vacuo, and in nitrogen. Samples were withdrawn at intervals and determinations made of the molecular weight, hexabromide number and iodine number. In separate experiments linseed oil was heated with S, Se, and Te. Samples withdrawn at intervals and the molecular weight determined. S, Se, and Te evolved as volatile sulfides, selenides, and tellurides were caught in ammoniacal $CdCl_2$ and the S, Se, and Te determined by titration.

Linolenic acid was prepared, converted to the hexabromide and heated with zinc dust and glycerol. The product is a glyceride. Its ultimate analysis corresponds to the mono-glyceride of linolenic acid. Constants were determined. This product was heated at 293° C. in air and in nitrogen. Samples were withdrawn at intervals and the molecular weight, hexabromide number and iodine number determined. The data thus determined indicate that at least two different types of reactions occur when linseed oil is heated. The data permit drawing some deductions as to the mechanism of these reactions.

A Comparison During Drying of the Acid Values and Iodine Numbers of Linseed Oil Containing Different Driers. W. Lloyd Evans, Paul E. Marling and Stewart E. Lower. In a series of experiments dealing with relationship between the acid value and iodine number of a drying film and use of different driers it was found:

1. The acid value of film is an increasing function of the time.
2. The iodine number of the film is a decreasing function of the time.
3. The acid value of the film is an increasing function of the concentration of the drier.
4. The iodine number of the film is a decreasing function of the concentration of the drier.

Studies of the Vitamin Potency of Cod Liver Oils. XXI. The Stimulation of Reproduction by Fat-Soluble Vitamins. A. D. Holmes, A. W. Doolittle and W. B. Moore. In preliminary studies, feeding a vitamin-rich cod liver oil to domestic fowl increased the number of chicks hatched. This investigation of thirty-two weeks' duration, was conducted to obtain data concerning factors associated with reproduction. A crude, vitamin-rich cod liver oil prepared by the steam process was fed to Rhode Island Red pullets. The experimental pens contained 75 birds which were fed ¼, ½, 1, and 2 cc. of oil per bird per day. Data were collected concerning egg yield, weight of egg, fertility, hatchibility, number of eggs containing blood spots, and mortality of chicks. Data were also collected concerning the effect of stimulated reproduction on the physical condition of the birds. It was found that supplementary vitamin feeding materially increased egg production, hatchibility, and viability of chicks; slightly increased fertility and weight of eggs; and decreased the number of eggs containing blood spots. The birds receiving the added vitamins maintained their body weight during the experiment and possessed a higher body resistance to disease, as indicated by a lower mortality than that of the control birds.

The Vitamin a Content of Pacific Coast Salmon Body Oils. Lyman C. Boynton and Roger Williams Truesdail. A quantitative study was made of the vitamin A content of the body oils of five species of Pacific Coast salmon. The chinook salmon was found to be the richest in this vitamin, followed in turn by sockeye, silver, humpback and chum varieties. Comparison was made with a sample of cod liver oil, one milligram per day of which was sufficient to induce a weight increase of approximately 25 grams in eight weeks in young rats whose store of vitamin A had been exhausted on an A-free basal diet. Young rats receiving 83 mg., 149 mg., and 226 mg. per day respectively of the body oil of chinook, sockeye, and silver salmon made an average growth of about 18 gr. over the eight weeks. Feeding 227 mg. per day of humpback oil furnished enough vitamin A for maintenance, while animals fed 221 mg. daily of chum salmon oil died before the expiration of the experimental period.

A correlation was found between the pigmentation of the oils and the vitamin A content. A notable exception was observed in the case of an unusually light specimen of chinook salmon which, however, is as a rule quite red. It was found to be richer in vitamin A than the more highly colored sockeye.

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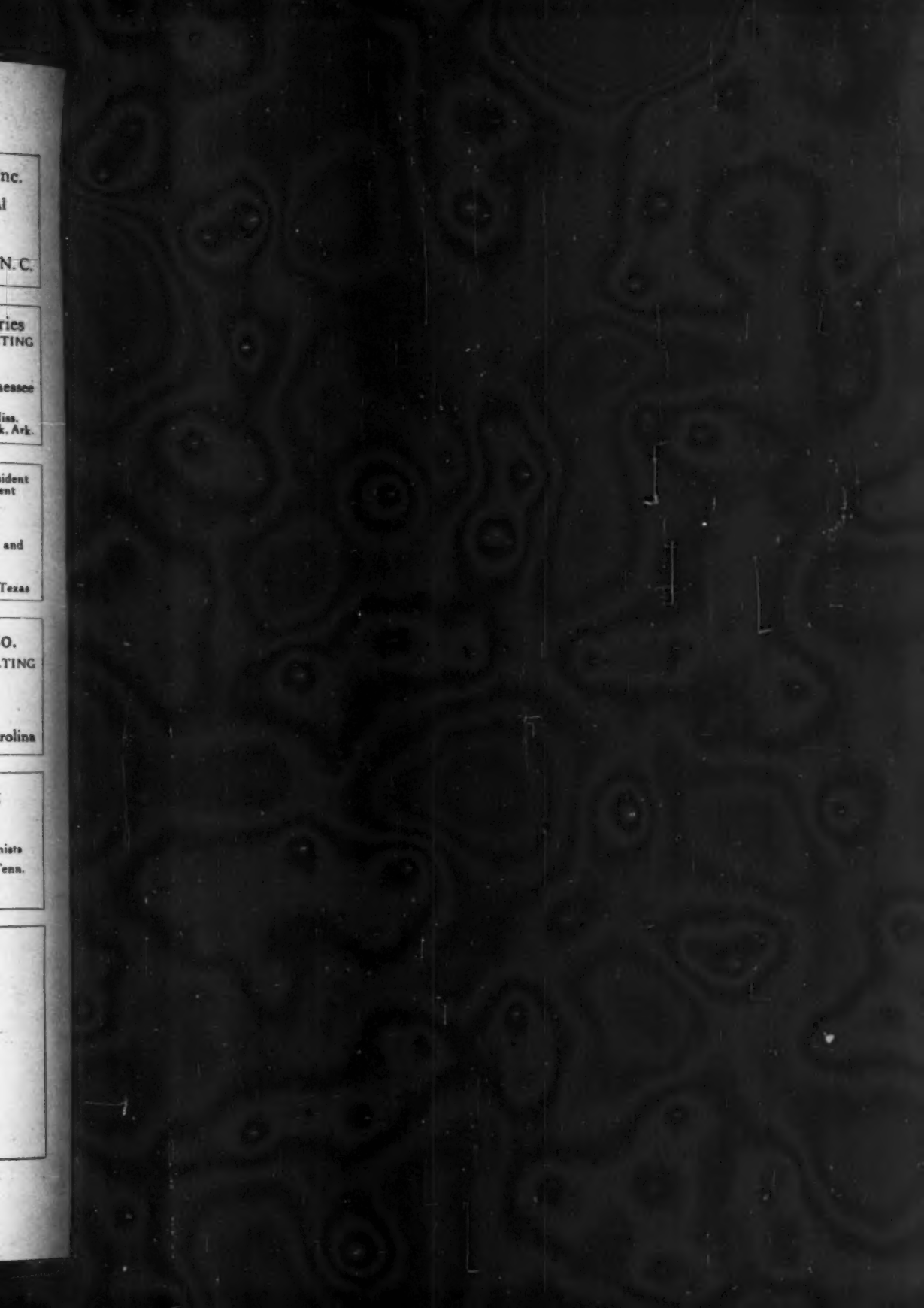
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